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Title: Actinide Analytical Chemistry Overview ICP Trace Element Analysis
(Including U, Np)

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Tandon, Lav

Intended for: Presentation

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Actinide Analytical Chemistry Overview

ICP Trace Element Analysis (Including U, Np)



Angela Olson, C-AAC

Lav Tandon, PT-1

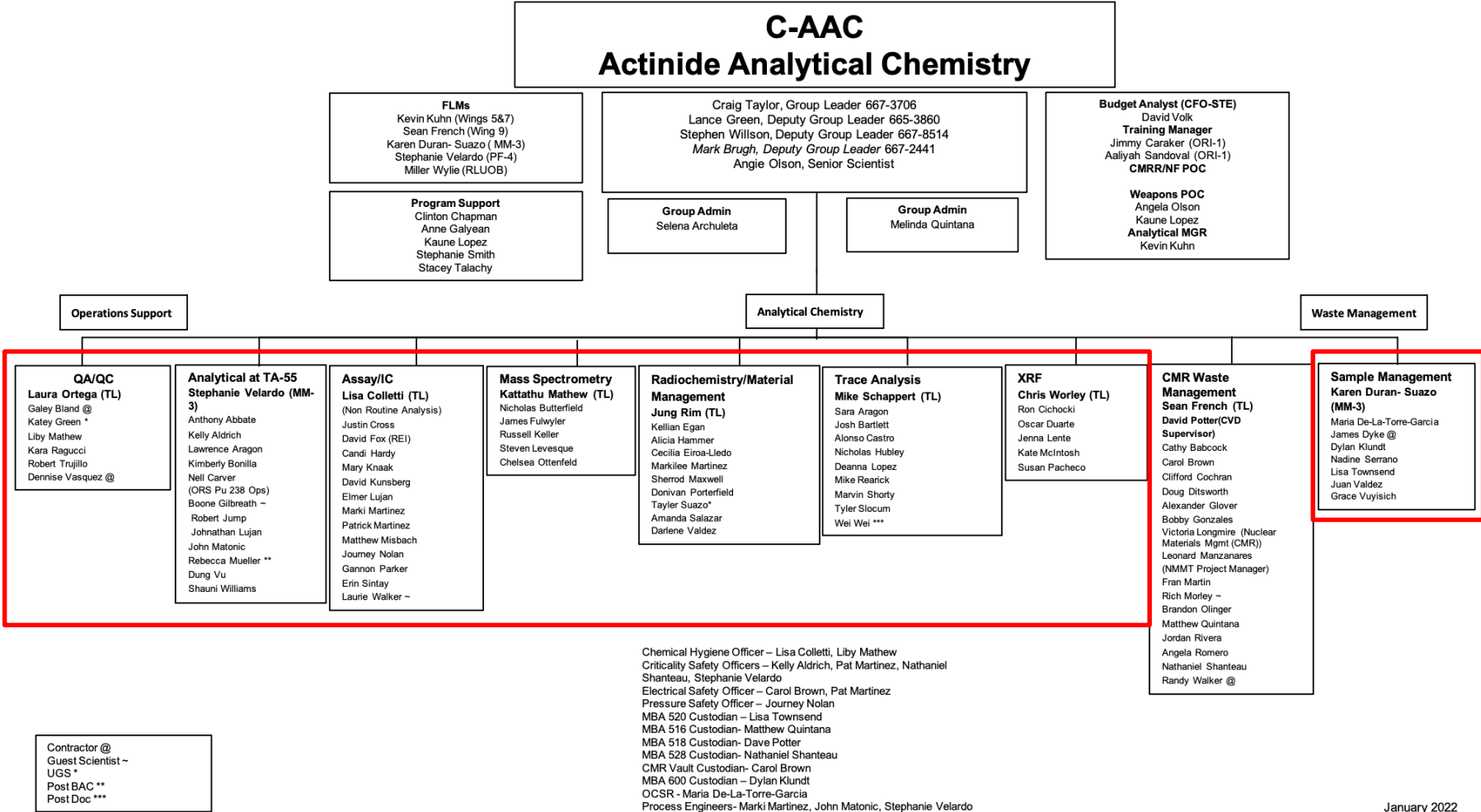
January 18, 2022

LA-UR-22-#####

Introduction

- Actinide Analytical Chemistry (C-AAC) at LANL has a full set of analytical chemistry capabilities to support the production mission
- AAC uses the Pu Metal Standards Exchange Program to validate and verify that the data quality objectives are met for Pu Sustainment
- This presentation describes
 - Analytical chemistry techniques used to address requirements
 - Analytical sample flow
 - Process overview

Organizational Structure



Summary of Analytical Techniques

Requirement	Analyte(s)	Primary Technique
Shall be Analyzed	Pu Isotopics	TIMS
	Pu Assay/Fe	Coulometry/Spectrophotometry
	Ga Assay	XRF
	Al, Be, Ca, Cr, Ni, Mg, Si, Ti	ICP-AES
	Ta, V, W	ICP-MS
	Am Assay	Radiochemistry
	Np Assay	Radiochemistry
	U Assay	TIMS
	C Concentration	Combustion/IR
Not Required	B, Cu, Mn, Zn	ICP-AES
	Cd, Pb, Sn, Th, Y, Zr	ICP-MS
	Cl, F Concentrations	Pyrohydrolysis/IC
	H, N, O Concentrations	Combustion/TCD

Summary of Analytical Techniques

Requirement	Analyte(s)	Primary Technique
Shall be Analyzed	Pu Isotopics	TIMS
	Pu Assay/Fe	Coulometry/Spectrophotometry
	Ga Assay	XRF
	Al, Be, Ca, Cr, Ni, Mg, Si, Ti	ICP-AES
	Ta, V, W	ICP-MS
	Am Assay	Radiochemistry
	Np Assay	Radiochemistry (ICP-MS)
	U Assay	TIMS (ICP-MS)
	C Concentration	Combustion/IR

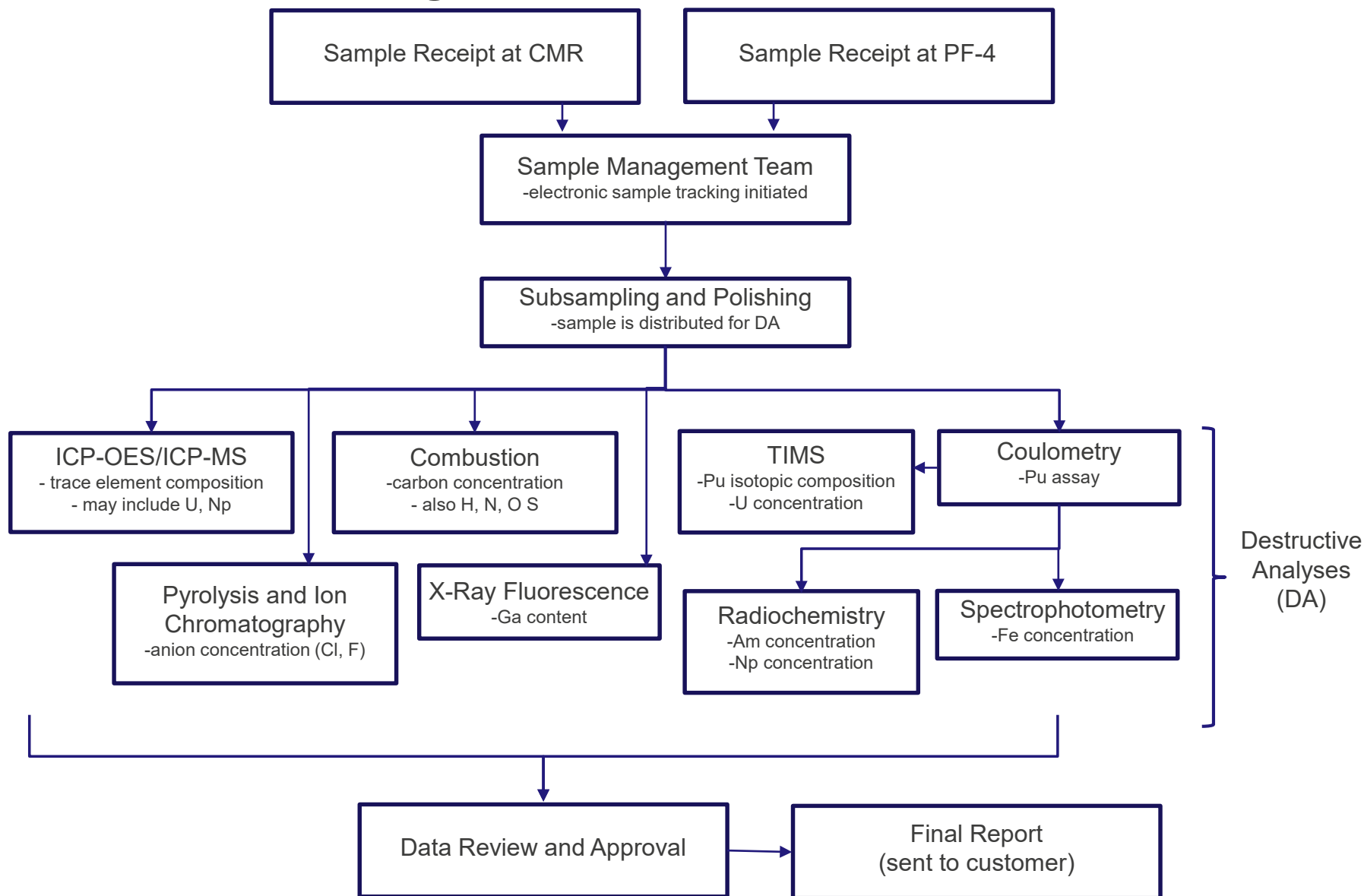
- Required analytes only
- Primary techniques and instruments only
- Consolidated techniques (e.g. ICP for U, Np)

Summary of Analytical Techniques

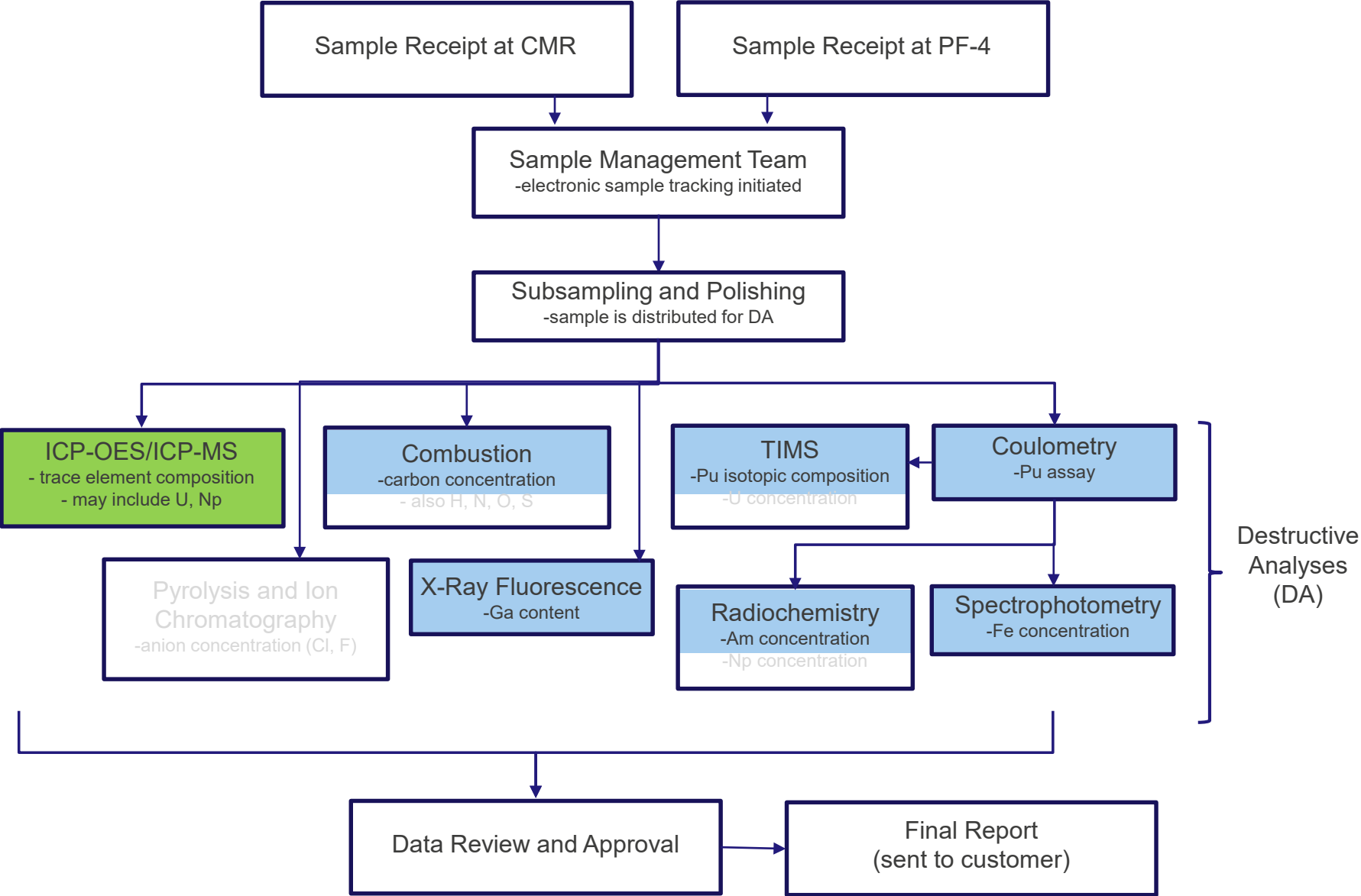
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Shall be Analyzed	Pu Isotopics	TIMS
	Pu Assay/Fe	Coulometry/Spectrophotometry
	Ga Assay	XRF
	Al, Be, Ca, Cr, Ni, Mg, Si, Ti	ICP-AES
	Ta, V, W	ICP-MS
	Am Assay	Radiochemistry
	Np Assay	Radiochemistry (ICP-MS)
	U Assay	TIMS (ICP-MS)
	C Concentration	Combustion/IR

- Required analytes only
- Primary techniques and instruments only
- Consolidated techniques (e.g. ICP for U, Np)

Example Analytical Sample Flow



Example Analytical Sample Flow

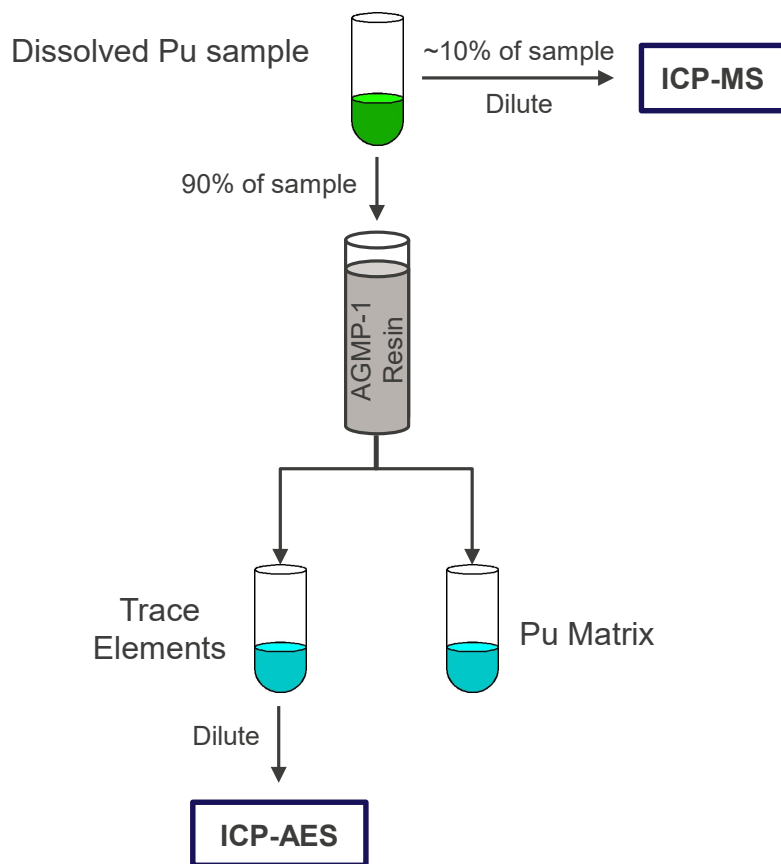


Primary Techniques and Instruments

- Used prior campaigns as basis for assigning primary techniques
- This EE includes primary ICP instruments only
 - Collected data for back-up instruments
 - Will compile and prepare data for back-up instruments to share at a future date

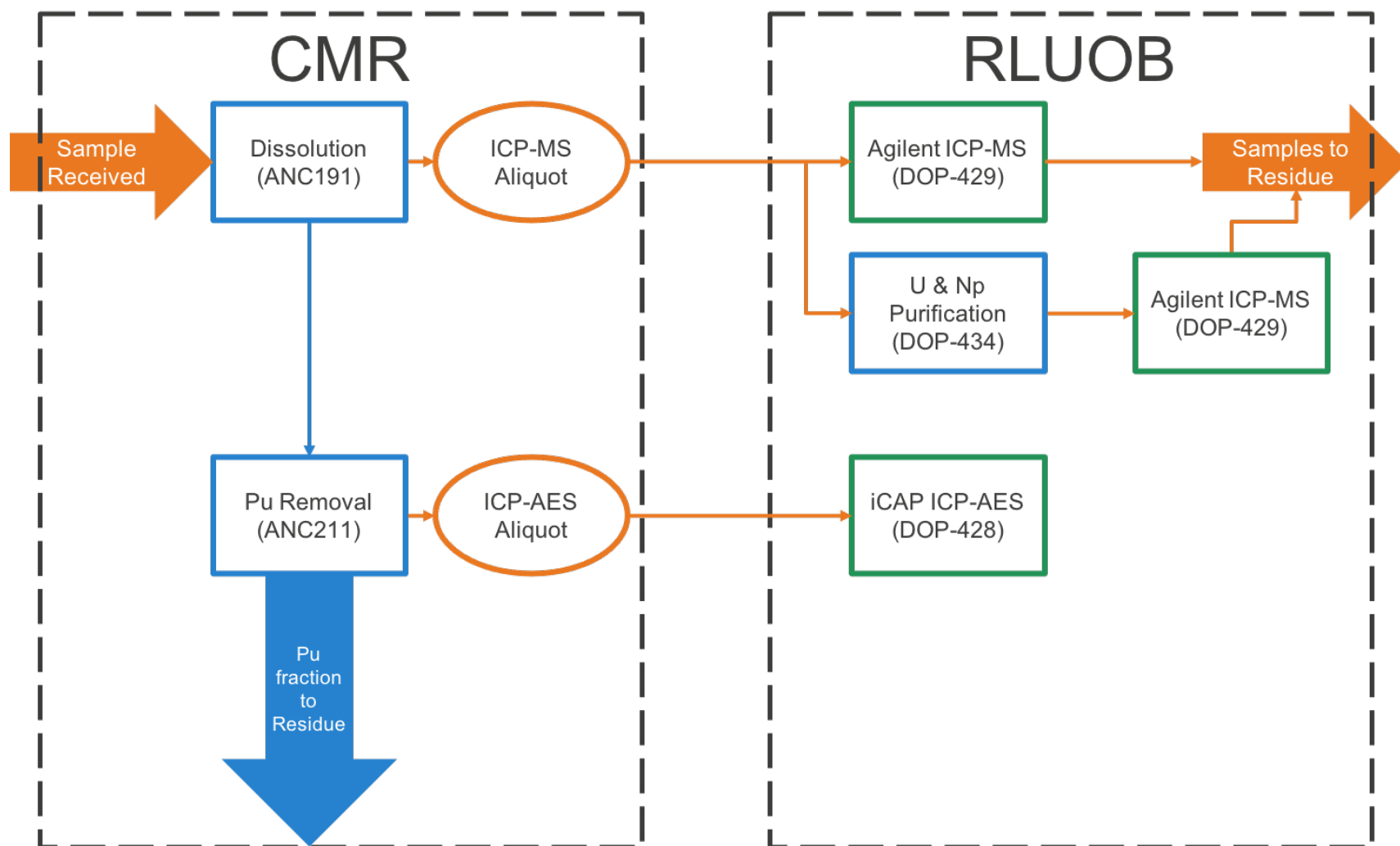
Analyte	Primary ICP Technique (Instrument)
Al	ICP-AES (iCAP 6500)
Be	
Ca	
Cr	
Ni	
Mg	
Si	
Ti	
Ta	ICP-MS (Agilent 7700)
V	
W	
U	
Np	

Trace Element Analysis



- Process is currently executed across 2 facilities (CMR and RLUOB)
- ICP-MS analyses typically carried out directly with dissolved sample (U is an exception)
- Pu matrix is removed for ICP-AES analysis

Trace Analysis Sample Flow



Engineering Evaluation Procedure Scope

- Trace Analysis process: 5 technical procedures

Location	Procedure(s)	Scope
CMR	ANC191, Plutonium-Metal Dissolution	Dissolution
	ANC211, U/Pu Removal for Impurity Assay	Trace metals sample preparation

Location	Procedure(s)	Scope
PF-400 (RLUOB)	CAAC-DOP-434, Sample Preparation for Trace Element Analysis at RLUOB	U/Np sample preparation
	CAAC-DOP-428, Thermo Scientific iCAP 6500/7600 ICP-AES Operation	ICP-AES analysis
	CAAC-DOP-429, Agilent 7700 ICP-MS Operation	ICP-MS analysis

Upcoming

Tabletop Reviews

- January 18:
 - Overview (this talk)
 - U/Np Discussion
 - Data Package Quality Review
- January 19: Procedure Reviews
- January 20: Data Reviews

Observations

- January 25: Trace Element Process Observation (CMR)
- January 26-27: Trace Element Process Observation (RLUOB)



EXTRA SLIDES






Introduction

- Actinide Analytical Chemistry (C-AAC) at LANL has a full set of analytical chemistry capabilities to support Pu Sustainment
- We use independent means to validate and verify that the highest data quality objectives are met for Pu Sustainment
 - All key analyses are validated on a regular schedule through the Plutonium Metals Exchange Program
- This presentation describes
 - Overview of analytical chemistry support for Pit Production
 - Analytical chemistry techniques used to address requirements
 - Analytical sample flow
 - Overview of processes

Analysis of Major, Minor, Trace Constituents

- **Major** – assay of plutonium (Pu), uranium (U), americium (Am), and neptunium (Np) with traceability to national standards
- **Minor** – from part-per-thousand to percent levels
 - critical to material performance
 - allows for interference corrections and mass balance
- **Trace** – from part-per-thousand to sub-part-per-million impurities

A large suite of elements with concentrations varying over several orders of magnitude (sub-ppm to %) are measured

-  Pu, U – Assay/ Fe Spectrophotometry
-  Thermal Ionization Mass Spectrometry
-  Radio Chemistry – Np, Am, Pu
-  X-ray Fluorescence – Ga
-  Cold Vapor Atomic Fluorescence

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Analytical Service Request

Attachment A, C-AAC Analytical Service Request Form

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Los Alamos National Laboratory
PO Box 1663
Los Alamos, NM 87544

Actinide Analytical Chemistry Group
Chemistry Division
Mail Stop G740
Telephone: 505-667-4087

Customer Information		ASR Rev#:	
Customer's Sample ID		Submitter's Name	
Date Sampled		Submitter's Z Number	
Date Submitted		Submitter's Telephone	
Sample Matrix		Submitter's Group	
Radioactive	<input type="checkbox"/> Yes <input type="checkbox"/> No	Requester's Name	
Radioactive Isotopes		Requester's Z-Number	
Project Name (Unclassified)		Requester's Telephone	
Cost Code (pc/ca/wp)		Requester's Group	
Report Distribution	Choose an item.	Send Report to MES	<input type="checkbox"/> Yes <input type="checkbox"/> No
Selecting Yes for Report to MES must be approved by C-AAC Sample Management			
Analytical Chemistry Information			
LabWare Sample ID:	Customer POC Review: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Yes, by: <u> </u> Z# or <u> </u> Z# or Other <u> </u> Z#		
Comments, Hazards, Special Handling & Regulatory Requirements for Analyses			
Comments:			
Hazards:		ICP-AES/ICP-MS Element List:	
Special Nuclear Materials Accountability (Sample Management Use Only)			
Accountable SNM: <input type="checkbox"/> Yes <input type="checkbox"/> No	Net Weight		
Project	Net Weight Units		
MASS NM Weight	NM Material Type		
Estimated NM Factor	Mass Enrichment %		
Sample Receipt Information			
Date/Time Received		Received By:	
		Initials/Z#	

Attachment A, C-AAC Analytical Service Request

(Page 2 of 3)

Analyses Required	
Actinide Assay	Trace Elements and Interstitials
<input type="checkbox"/> Dissolution and Aliquoting (DISS-ASSAY) <input type="checkbox"/> CMR <input type="checkbox"/> RLUOB <input type="checkbox"/> TA55	
<input type="checkbox"/> Radiochemistry Survey (RC-SURVEY) (RD-GAMMA)	<input type="checkbox"/> ICP Trace Elements (PSSUR-S) (ICP-MS-S) & (ICP-AES-S)
<input type="checkbox"/> Pu Assay by Coulometry (PACPU) (PU-ASSAY) Choose an item.	<input type="checkbox"/> Fe by spectrophotometry (PA-FE) (FE)
<input type="checkbox"/> U Assay by Davies-Gray (PAUDG) (U-ASSAY)	<input type="checkbox"/> Gallium analysis by XRF (XRF-GA) Choose an item.
<input type="checkbox"/> Np Assay by Coulometry (PACPU) (NP-ASSAY)	<input type="checkbox"/> Gallium and Uranium analysis by XRF (XRF-GA-U)
<input type="checkbox"/> Pu Assay by IDMS (MSPUID-S) (MS-PU-ID-S) Choose an item.	<input type="checkbox"/> Gallium analysis by IDMS (MS-GA-ID-S) Choose an item.
<input type="checkbox"/> U Assay by IDMS (MSUID-S) (MS-U-ID-S)	<input type="checkbox"/> Carbon by Interstitial Gas Analysis (IC-C) (CARBON)
<input type="checkbox"/> Am Assay by IDMS (MSUID-S) (MS-AM-ID-S)	<input type="checkbox"/> Oxygen by Interstitial Gas Analysis (IC-O) (OXYGEN)
<input type="checkbox"/> Th Assay by IDMS (MSUID-S) (MS-TH-ID-S)	<input type="checkbox"/> Hydrogen by Interstitial Gas Analysis (IC-H) (HYDROGEN)
<input type="checkbox"/> Pu Assay by Spectrophotometry (PU-SPEC-ASSAY)	<input type="checkbox"/> Sulfur by Interstitial Gas Analysis (IC-S) (SULFUR)
<input type="checkbox"/> Pu Assay by Titration (PU-ASSAY-TITRATION)	<input type="checkbox"/> Nitrogen (IC-KJELDAHL & IC-NITROGEN-S)
<input type="checkbox"/> Alpha Spectroscopy Survey (RD-ALPHA-SPEC)	<input type="checkbox"/> Hg by Cold Vapor Atomic Fluorescence (CVAF)
<input type="checkbox"/> Plutonium by Radiochemistry, weight of sample (RC-PU-WT)	<input type="checkbox"/> Perchlorates (IC-CLO4) (PERCHLORATE)
<input type="checkbox"/> Americium by Radiochemistry, weight of sample (RC-AM-WT)	<input type="checkbox"/> Chloride and Fluoride by IC (PYRO-F-CL & IC-ANIONS-S)
<input type="checkbox"/> Neptunium by Radiochemistry, weight of sample (RC-NP-WT)	<input type="checkbox"/> Anion Analysis by Ion Chromatography (IC-ANIONS)
<input type="checkbox"/> Radiochem of Pu-238 for Am-241, Pu-236, U-234 (RC-PU-238)	<input type="checkbox"/> Silicon by Colorimetric Analysis (SILICON)
<input type="checkbox"/> Pu, Am-241 by Radiochemistry, liquid sample (RC-PU-AM-L)	<input type="checkbox"/> Loss on Ignition (LOI)
Isotopic Analyses	Bulk Measurements
<input type="checkbox"/> Pu Isotopes (MS-PU-ISO-S)	<input type="checkbox"/> Density by Pycnometry (DENSITY)
<input type="checkbox"/> U Isotopes (MS-U-ISO-S)	<input type="checkbox"/> TCLP Analysis (PS_TCLP)
<input type="checkbox"/> Am 241 Isotopes (MS-AM241-RATIO)	<input type="checkbox"/> DC Arc Analysis (PS-DC-ARC)
<input type="checkbox"/> Am 243 Isotopes (MS-AM243-RATIO)	<input type="checkbox"/> SEM Analysis (RD-SEM)
<input type="checkbox"/> Np 237 Isotopes (MS-NP237-ATOM-RATIO)	<input type="checkbox"/> X-ray Diffraction Analysis (RD-XRD)
<input type="checkbox"/> Th 232 Isotopes (MS-TH232-ATOM-RATIO)	<input type="checkbox"/> Quantitative XRF <input type="checkbox"/> WD (XRF-QUANTITATIVE)
<input type="checkbox"/> Age by Specific Isotope Ratio: (MS-AGE-various) Choose an item.	<input type="checkbox"/> Semi-Quantitative XRF <input type="checkbox"/> WD (XRF-SEMI-QUANT)
<input type="checkbox"/> Gamma Isotopic Analysis using FRAM (FRAM)	<input type="checkbox"/> Qualitative XRF <input type="checkbox"/> WD (XRF-QUALITATIVE)
<input type="checkbox"/> Am and Np Gamma Analysis using FRAM (FRAM-AM-NP)	<input type="checkbox"/> Molarity determined by Acid-Base Titration (ACID-BASE)
TA-55 Specific Analyses	
<input type="checkbox"/> Carbon (Carbon@TA55)	

Analytical Service Request

Attachment A, C-AAC Analytical Service Request Form

(Page 1 of 3)

Los Alamos National Laboratory
PO Box 1663
Los Alamos, NM 87544

Actinide Analytical Chemistry Group
Chemistry Division
Mail Stop G740
Telephone: 505-667-4087

Customer Information		ASR Rev#:	
Customer's Sample ID		Submitter's Name	
Date Sampled		Submitter's Z Number	
Date Submitted		Submitter's Telephone	
Sample Matrix		Submitter's Group	
Radioactive	<input type="checkbox"/> Yes <input type="checkbox"/> No	Requester's Name	
Radioactive Isotopes		Requester's Z-Number	
Project Name (Unclassified)		Requester's Telephone	
Cost Code (pc/ca/wp)		Requester's Group	
Report Distribution	Choose an item.	Send Report to MES	<input type="checkbox"/> Yes <input type="checkbox"/> No
Selecting Yes for Report to MES must be approved by C-AAC Sample Management			
Analytical Chemistry Information			
LabWare Sample ID:		Customer POC Review: <input type="checkbox"/> Yes <input type="checkbox"/> No	
		Yes, by: <u> </u> Z# or <u> </u> Z# or <u> </u> Z#	
Regulatory Requirements for Analyses			
Comments:			
Hazards:		ICP-AES/ICP-MS Element List:	
Special Nuclear Materials Accountability (Sample Management Use Only)			
Accountable SNM: <input type="checkbox"/> Yes <input type="checkbox"/> No		Net Weight	
Project		Net Weight Units	
MASS NM Weight		NM Material Type	
Estimated NM Factor		Mass Enrichment %	
Sample Receipt Information			
Date/Time Received		Received By:	
		Initials/Z#	

Attachment A, C-AAC Analytical Service Request
(Page 2 of 3)

Analyses Required	
Actinide Assay	Trace Elements and Interstitials
<input type="checkbox"/> Dissolution and Aliquoting (DISS-ASSAY) <input type="checkbox"/> CMR <input type="checkbox"/> RLUOB <input type="checkbox"/> TA55	
<input type="checkbox"/> Radiochemistry Survey (RC-SURVEY) (RD-GAMMA)	<input type="checkbox"/> ICP Trace Elements (PSSUR-S) (ICP-MS-S) & (ICP-AES-S)
<input type="checkbox"/> Pu Assay by Coulometry (PACPU) (PU-ASSAY) Choose an item.	<input type="checkbox"/> Fe by spectrophotometry (PA-FE) (FE)
<input type="checkbox"/> U Assay by Davies-Gray (PAUDG) (U-ASSAY)	<input type="checkbox"/> Gallium analysis by XRF (XRF-GA) Choose an item.
<input type="checkbox"/> Np Assay by Coulometry (PACPU) (NP-ASSAY)	<input type="checkbox"/> Gallium and Uranium analysis by XRF (XRF-GA-U)
<input type="checkbox"/> Pu Assay by IDMS (MSPUID-S) (MS-PU-ID-S) Choose an item.	<input type="checkbox"/> Gallium analysis by IDMS (MS-GA-ID-S) Choose an item.
<input type="checkbox"/> U Assay by IDMS (MSUID-S) (MS-U-ID-S)	<input type="checkbox"/> Carbon by Interstitial Gas Analysis (IC-C) (CARBON)
<input type="checkbox"/> Am Assay by IDMS (MSUID-S) (MS-AM-ID-S)	<input type="checkbox"/> Oxygen by Interstitial Gas Analysis (IC-O) (OXYGEN)
<input type="checkbox"/> Th Assay by IDMS (MSUID-S) (MS-TH-ID-S)	<input type="checkbox"/> Hydrogen by Interstitial Gas Analysis (IC-H) (HYDROGEN)
<input type="checkbox"/> Pu Assay by Spectrophotometry (PU-SPEC-ASSAY)	<input type="checkbox"/> Sulfur by Interstitial Gas Analysis (IC-S) (SULFUR)
<input type="checkbox"/> Pu Assay by Titration (PU-ASSAY-TITRATION)	<input type="checkbox"/> Nitrogen (IC-KJELDAHL & IC-NITROGEN-S)
<input type="checkbox"/> Alpha Spectroscopy Survey (RD-ALPHA-SPEC)	<input type="checkbox"/> Hg by Cold Vapor Atomic Fluorescence (CVAF)
<input type="checkbox"/> Plutonium by Radiochemistry, weight of sample (RC-PU-WT)	<input type="checkbox"/> Perchlorates (IC-CLO4) (PERCHLORATE)
<input type="checkbox"/> Americium by Radiochemistry, weight of sample (RC-AM-WT)	<input type="checkbox"/> Chloride and Fluoride by IC (PYRO-F-CL & IC-ANIONS-S)
<input type="checkbox"/> Neptunium by Radiochemistry, weight of sample (RC-NP-WT)	<input type="checkbox"/> Anion Analysis by Ion Chromatography (IC-ANIONS)
<input type="checkbox"/> Radiochem of Pu-238 for Am-241, Pu-236, U-234 (RC-PU-238)	<input type="checkbox"/> Silicon by Colorimetric Analysis (SILICON)
<input type="checkbox"/> Pu, Am-241 by Radiochemistry, liquid sample (RC-PU-AM-L)	<input type="checkbox"/> Loss on Ignition (LOI)
Isotopic Analyses	Bulk Measurements
<input type="checkbox"/> Pu Isotopes (MS-PU-ISO-S)	<input type="checkbox"/> Density by Pycnometry (DENSITY)
<input type="checkbox"/> U Isotopes (MS-U-ISO-S)	<input type="checkbox"/> TCLP Analysis (PS_TCLP)
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<input type="checkbox"/> Am 243 Isotopes (MS-AM243-RATIO)	<input type="checkbox"/> SEM Analysis (RD-SEM)
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<input type="checkbox"/> Th 232 Isotopes (MS-TH232-ATOM-RATIO)	<input type="checkbox"/> Quantitative XRF <input type="checkbox"/> WD (XRF-QUANTITATIVE)
<input type="checkbox"/> Age by Specific Isotope Ratio: (MS-AGE-various) Choose an item.	<input type="checkbox"/> Semi-Quantitative XRF <input type="checkbox"/> WD (XRF-SEMI-QUANT)
<input type="checkbox"/> Gamma Isotopic Analysis using FRAM (FRAM)	<input type="checkbox"/> Qualitative XRF <input type="checkbox"/> WD (XRF-QUALITATIVE)
<input type="checkbox"/> Am and Np Gamma Analysis using FRAM (FRAM-AM-NP)	<input type="checkbox"/> Molarity determined by Acid-Base Titration (ACID-BASE)
TA-55 Specific Analyses	
<input type="checkbox"/> Carbon (Carbon@TA55)	

Unique Sample Identifiers

Attachment A, C-AAC Analytical Service Request Form

(Page 1 of 3)

Los Alamos National Laboratory
PO Box 1663
Los Alamos, NM 87544

Actinide Analytical Chemistry Group
Chemistry Division
Mail Stop G740
Telephone: 505-667-4087

Customer Information		ASR Rev#:	
Customer's Sample ID		Submitter's Name	
Date Sampled		Submitter's Z Number	
Date Submitted		Submitter's Telephone	
Sample Matrix		Submitter's Request	
Radioactive	<input type="checkbox"/> Yes <input type="checkbox"/> No	Request Number	
Radioactive Isotopes		Request Telephone	
Project Name (Unclassified)		Requester	
Cost Code (pc/ca/wp)		Send Report MES	
Report Distribution	Choose an item.		
Selecting Yes for Report to MES must be approved by C-AAC			
Analytical Chemistry Information			
LabWare Sample ID:		Customer's	
		<input type="checkbox"/> Yes, by: Z# 01 Z# 01	
		Other Z#	
Comments, Hazards, Special Handling & Regulatory Requirements for Analyses			
Comments:			
Hazards:		ICP-AES/ICP-MS Element List:	
Special Nuclear Materials Accountability (Sample Management Use Only)			
Accountable SNM: <input type="checkbox"/> Yes <input type="checkbox"/> No	Net Weight		
Project	Net Weight Units		
MASS NM Weight	NM Material Type		
Estimated NM Factor	Mass Enrichment %		
Sample Receipt Information			
Date/Time Received		Received By:	
		Initials/Z#	

✓ Samples are tracked electronically throughout each process (Labware)

LabWare Sample ID:
29353 A29353-0

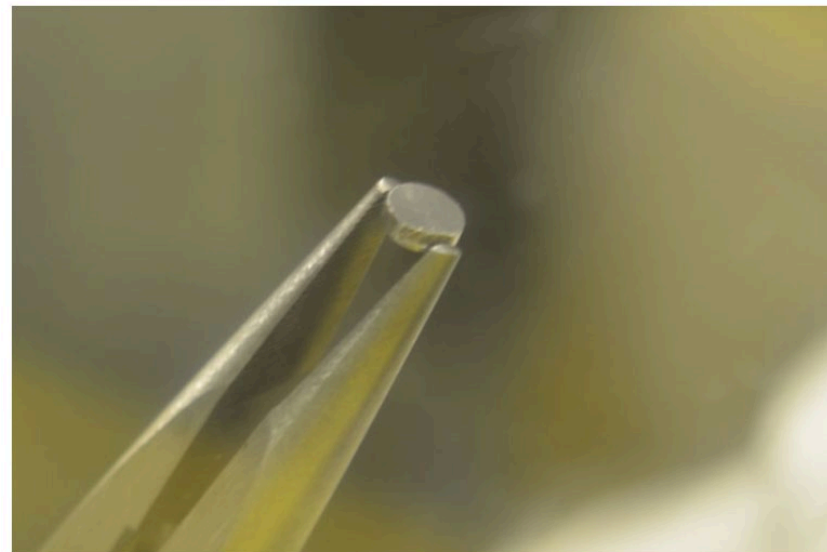
Attachment A, C-AAC Analytical Service Request
(Page 2 of 3)

Analyses Required	
Actinide Assay	Trace Elements and Interstitials
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<input type="checkbox"/> Radiochemistry Survey (RC-SURVEY) (RD-GAMMA)	<input type="checkbox"/> Fe by spectrophotometry (PA-FE) (FE)
<input type="checkbox"/> U Assay by IDMS (MSUID-S) (MS-U-ID-S)	<input type="checkbox"/> Gallium analysis by XRF (XRF-GA) Choose an item.
	<input type="checkbox"/> Gallium and Uranium analysis by XRF (XRF-GA-U)
	<input type="checkbox"/> Gallium analysis by IDMS (MS-GA-ID-S) Choose an item.
	<input type="checkbox"/> Carbon by Interstitial Gas Analysis (IC-C) (CARBON)
	<input type="checkbox"/> Oxygen by Interstitial Gas Analysis (IC-O) (OXYGEN)
	<input type="checkbox"/> Hydrogen by Interstitial Gas Analysis (IC-H) (HYDROGEN)
	<input type="checkbox"/> Sulfur by Interstitial Gas Analysis (IC-S) (SULFUR)
	<input type="checkbox"/> Nitrogen (IC-KJELDAHL & IC-NITROGEN-S)
	<input type="checkbox"/> Hg by Cold Vapor Atomic Fluorescence (CVAF)
<input type="checkbox"/> Plutonium by Radiochemistry, weight of sample (RC-PU-WT)	<input type="checkbox"/> Perchlorates (IC-CLO4) (PERCHLORATE)
<input type="checkbox"/> Americium by Radiochemistry, weight of sample (RC-AM-WT)	<input type="checkbox"/> Chloride and Fluoride by IC (PYRO-F-CL & IC-ANIONS-S)
<input type="checkbox"/> Neptunium by Radiochemistry, weight of sample (RC-NP-WT)	<input type="checkbox"/> Anion Analysis by Ion Chromatography (IC-ANIONS)
<input type="checkbox"/> Radiochem of Pu-238 for Am-241, Pu-236, U-234 (RC-PU-238)	<input type="checkbox"/> Silicon by Colorimetric Analysis (SILICON)
<input type="checkbox"/> Pu, Am-241 by Radiochemistry, liquid sample (RC-PU-AM-L)	<input type="checkbox"/> Loss on Ignition (LOI)
Isotopic Analyses	Bulk Measurements
<input type="checkbox"/> Pu Isotopes (MS-PU-ISO-S)	<input type="checkbox"/> Density by Pycnometry (DENSITY)
<input type="checkbox"/> U Isotopes (MS-U-ISO-S)	<input type="checkbox"/> TCLP Analysis (PS_TCLP)
<input type="checkbox"/> Am 241 Isotopes (MS-AM241-RATIO)	<input type="checkbox"/> DC Arc Analysis (PS-DC-ARC)
<input type="checkbox"/> Am 243 Isotopes (MS-AM243-RATIO)	<input type="checkbox"/> SEM Analysis (RD-SEM)
<input type="checkbox"/> Np 237 Isotopes (MS-NP237-ATOM-RATIO)	<input type="checkbox"/> X-ray Diffraction Analysis (RD-XRD)
<input type="checkbox"/> Th 232 Isotopes (MS-TH232-ATOM-RATIO)	<input type="checkbox"/> Quantitative XRF <input type="checkbox"/> WD (XRF-QUANTITATIVE)
<input type="checkbox"/> Age by Specific Isotope Ratio: (MS-AGE-various) Choose an item.	<input type="checkbox"/> Semi-Quantitative XRF <input type="checkbox"/> WD (XRF-SEMI-QUANT)
<input type="checkbox"/> Gamma Isotopic Analysis using FRAM (FRAM)	<input type="checkbox"/> Qualitative XRF <input type="checkbox"/> WD (XRF-QUALITATIVE)
<input type="checkbox"/> Am and Np Gamma Analysis using FRAM (FRAM-AM-NP)	<input type="checkbox"/> Molarity determined by Acid-Base Titration (ACID-BASE)
TA-55 Specific Analyses	
<input type="checkbox"/> Carbon (Carbon@TA55)	

Sample Polishing



unpolished encapsulated Pu metal



polished Pu metal

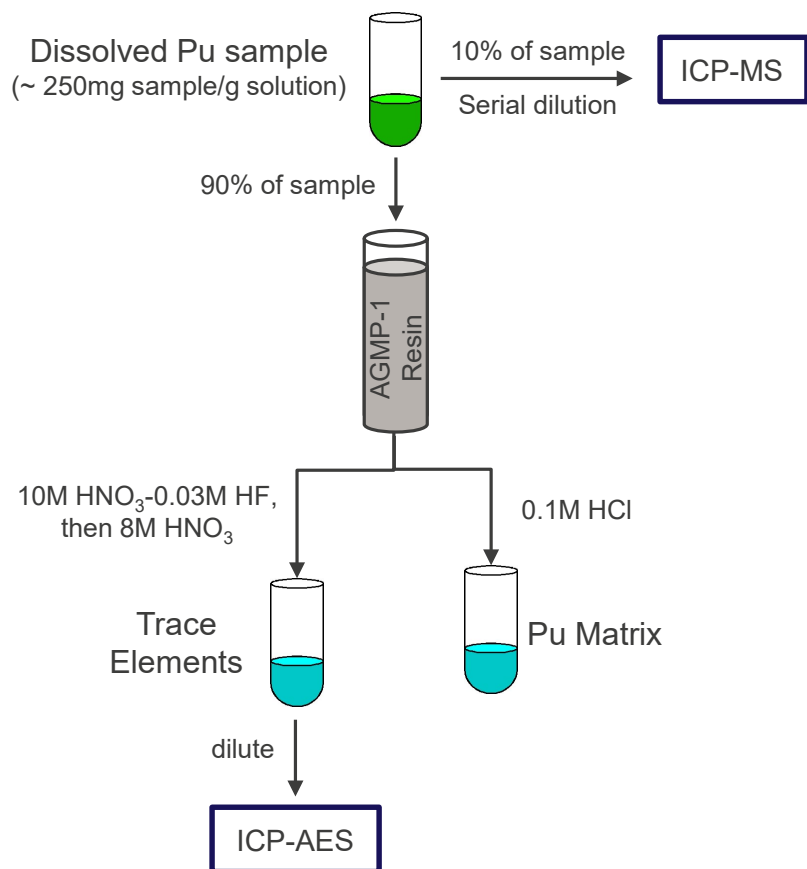
- Polishing is critical for high accuracy, high precision analyses
 - Wire brush is used to remove surface oxide layer
 - Performed in an inert atmosphere glovebox

Sample Size Requirements

- Quality control standards and blanks are analyzed in parallel to samples where applicable
- 5 g sample size is preferred, and generally allows for recuts
- Samples are typically analyzed in duplicate (sometimes triplicate)

Analysis Method(s)	Individual Cut Size (mg)	# of Cuts	Total Sample Size (mg)
Pu Assay/Fe	475-499	2	950-998
U Concentration	(small portion from assay cut)	2	<5
Pu Isotopics	(small portion from assay cut)	2	<5
Ga (XRF)	475-499	2	950-998
Am, Np Concentration	(80-120; from assay cut)	2	160-240
Carbon	400-499	2	800-998
Trace Element Concentrations	235-275 + 235-275 per batch	3	705-825
Cl, F Concentrations	475-499	2	950-998
TOTAL			4.52 - 5.06 g

Trace Elements



- Analyte list varies depending on specification
- Pu matrix does not interfere with ICP-MS analysis
- Pu matrix interferes with ICP-AES analysis
 - Pu matrix is removed using AGMP-1 Resin



Primary Techniques and Backups

Required Analytes

Analyte	Primary ICP Technique (Instrument)	Back-up Technique
Al	ICP-AES (iCAP 6500)	ICP-MS
Be		ICP-MS
Ca		n/a
Cr		ICP-MS
Ni		ICP-MS
Mg		ICP-MS
Si		Spectrophotometry
Ti		ICP-MS
Ta	ICP-MS (Agilent 7700)	ICP-AES
V		ICP-AES
W		ICP-AES
U		TIMS
Np		Radiochemistry

Primary Techniques and Backups

Not Required

Analyte	Primary ICP Technique (Instrument)	Back-up Technique
B	ICP-AES	ICP-MS
Cu		ICP-MS
Mn		ICP-MS
Zn		ICP-MS
Cd	ICP-MS	ICP-AES
Pb		ICP-AES
Sn		ICP-AES
Th		n/a
Y		ICP-AES
Zr		ICP-AES

Overview of 2018 ER Ring EE

- From May-July 2018, an EE was completed in support of the FY18 ER Ring milestone
- The LLNL Design Agency evaluated Analytical Chemistry procedures and practices
- Evaluators included:
 - LLNL Product Realization Team (PRT) Lead
 - LLNL Quality Engineer
 - LLNL Technical Experts
- EE was conducted in two phases
 - Phase 1: Tabletop (24 chemistry procedures; 11 processes)
 - Phase 2: Observational (6 chemistry procedures; 3 processes)

Scope of 2018 ER Ring EE

Phase 1 – Tabletop

- Technical discussion completed for each procedure
- Modifications were suggested and incorporated

Phase 2 – Observational

- Walk-down of 3 processes:
 - Pu Assay by Coulometry
 - Fe by Spectrophotometry
 - Trace Elements Analysis by ICP-AES
- 6 procedures covered:
 - Sample handling, tracking, dissolution, splitting/cutting, blanks (instrument, process, and reagent), quality control samples, replicate analysis, data analysis, data entry
- Additionally:
 - Training, document control, M&TE, records, QA protocols
 - MDLs, coulometry blanks

Process Qualification Plans (PQPs)

- PQPs were developed (1 for each process)
 - Qualification of analyte, instrument, and method
 - Leverages existing historical data, where possible
 - Methods have been continually validated since last campaign through the Pu Metal Standards Exchange Program (PMSEP)
- Purpose
 - Establish standards that will be used for measuring each analyte in Pu metal
 - Demonstrate each process through supporting data
 - Statistical evaluation to meet the product related requirements
 - Assure that the instrument and respective analytical methods meet the accuracy, precision, and uncertainty expectations
- Demonstration of statistical control and repeatability is key

Process Qualification Plans (PQPs)

- Scope
 - The following aspects were captured: sample preparation, chemical analysis, instrument techniques, data collected, statistical analyses of results
 - Data were collected using certified (traceable) standards, PMSEP materials, quality control data, and development samples from the manufacturing team
 - Data collected have been used to determine accuracy, precision, uncertainty, and possible biases/trends
 - Experiments define inherent repeatability and reproducibility
- A Process Qualification Report (PQR) will be prepared, summarizing experimental results